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**Datasheet for the decision  
of 5 February 2010**

**Case Number:** T 0297/07 - 3.3.05

**Application Number:** 01996519.3

**Publication Number:** 1343739

**IPC:** C05C 13/00

**Language of the proceedings:** EN

**Title of invention:**  
AMMONIUM SULFATE NITRATE

**Patentee:**  
Honeywell International Inc.

**Opponent:**  
YARA INTERNATIONAL ASA

**Headword:**  
Ammonium Sulfate Nitrate/HONEYWELL INT. INC.

**Relevant legal provisions:**  
EPC Art. 54, 56

**Relevant legal provisions (EPC 1973):**  
-

**Keyword:**  
"Novelty (yes)"  
"Inventive step (yes) - evidence for the technical solution in  
the patent in suit"

**Decisions cited:**  
-

**Catchword:**  
-



Case Number: T 0297/07 - 3.3.05

**DECISION**  
of the Technical Board of Appeal 3.3.05  
of 5 February 2010

**Appellant:** YARA INTERNATIONAL ASA  
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**Decision under appeal:** Decision of the Opposition Division of the  
European Patent Office posted 20 December 2006  
rejecting the opposition filed against European  
patent No. 1343739 pursuant to Article 102(2)  
EPC.

**Composition of the Board:**

**Chairman:** G. Raths  
**Members:** H. Engl  
H. Preglau

## Summary of Facts and Submissions

I. This appeal lies from the decision of the opposition division posted on 20 December 2006 to reject the opposition against European patent EP-B-1 343 739.

II. The independent claims of the patent as granted are worded as follows:

"1. , [sic] A non-explosive ammonium sulfate nitrate composite material comprising by x-ray diffraction analysis:

about 14 wt.% to about 35 wt.% ammonium sulfate  $((\text{NH}_4)_2\text{SO}_4)$ ;

about 60 wt.% to about 85 wt.%  $(\text{NH}_4)_2\text{SO}_4 \cdot 2(\text{NH}_4\text{NO}_3)$  double salt; and to about 5 wt.% in combined total  $(\text{NH}_4)_2\text{SO}_4 \cdot 3(\text{NH}_4\text{NO}_3)$  double salt and ammonium nitrate  $(\text{NH}_4\text{NO}_3)$ ; [sic]"

"2. , [sic] A non-explosive ammonium sulfate nitrate composite material, by x-ray diffraction analysis, consisting essentially of:

about 14 wt.% to about 35 wt.% ammonium sulfate  $((\text{NH}_4)_2\text{SO}_4)$ ;

about 60 wt.% to about 85 wt.%  $(\text{NH}_4)_2\text{SO}_4 \cdot 2(\text{NH}_4\text{NO}_3)$  double salt; and 0 to about 5 wt.% in combined total  $(\text{NH}_4)_2\text{SO}_4 \cdot 3(\text{NH}_4\text{NO}_3)$  double salt and ammonium nitrate  $(\text{NH}_4\text{NO}_3)$ ."

Claims 3 to 5 represent particular embodiments of the subject matter of claims 1 and 2.

"6. A method for the production of an ammonium sulfate nitrate composite material comprising the steps of:

- (a) charging materials comprising ammonium sulfate particles, ammonium nitrate and water to a melting device, wherein the molar ratio of ammonium sulfate to ammonium nitrate is about 0.9:1 to about 1.1:1 and the water is more than 2 wt.% to about 10 wt.% of the charged materials;
- (b) melting the ammonium nitrate and dissolving at least a portion of the ammonium sulfate particles at a temperature of about 180 °C to about 210 °C;
- (c) reacting the charged materials at a temperature of about 180 °C to about 210°C; and
- (d) solidifying the product at a cooling rate of at least about 100 °C/min."

Claims 7 to 19 represent particular embodiments of the method of claim 6.

"20. An ammonium sulfate nitrate composite material prepared by the method of claims 6 or 7."

III. The following documents were cited in the opposition procedure:

- D1: H.-H. Emons et al.: "Untersuchungen am System Ammonsulfat - Ammonnitrat - Wasser", Wissenschaftl. Zeitschr. 10, (1968) Heft 2-3, pages 102-106.
- D2: J. P. Smith et al.: "Crystallographic Properties of the Ammonium Nitrate-Sulfates  $3\text{NH}_4\text{NO}_3 \cdot (\text{NH}_4)_2\text{SO}_4$  and  $2\text{NH}_4\text{NO}_3 \cdot (\text{NH}_4)_2\text{SO}_4$ " Agricultural Food and Chemistry, (1962) Vol. 10, No. 1, pages 77 and 78.
- D3: I.S.M.A., Réunion Technique, Prague, Tchécoslovaquie, 23 - 27 September 1974,

Association Internationale des Fabricants de Superphosphate et d'Engrais Composés: J. Ando, *"Caking and Degradation of Granular Compound Fertilizers Containing Nitrates and Sulfates"*, pages 2-1 to 2-9.

- D4: W. F. Linke, *"Solubilities of Inorganic and Metal-Organic Compounds"*, (1965) Vol. II, pages 712 to 713.
- D5: The New Oxford Dictionary, Oxford University Press, 1999, page 377.
- D6: L. Medard, *Les Explosifs Occasionnels*, (1979) Vol. 2, Chapter 25.3: *"Le Sulfonitrate d'Ammonium"*, pages 662-663.
- D7: The International Fertiliser Society, Proceedings No. 124, (1971), G. Perbal: *"The Thermal Stability of Fertilisers Containing Ammonium Nitrate"*, pages 3 to 15.
- D8: Publication of the British Sulfur Corp. Ltd., London, UK (1970): *"Ammonium Nitrate - Supply/Demand 1957-1977"*, pages 32 to 35.
- D9: US-A-2 762 699
- D10: Kirk - Othmer, *Concise Encyclopedia of Chemical Technology*, John Wiley & Sons, New York, 1985, pages 307 - 309.

IV. The opposition division held that documents D1 to D5 failed to disclose individually and unambiguously the

proportions of ammonium sulphate (AS) and ammonium sulphate (AS) / ammonium nitrate (AN) 1:2 double salt claimed in the opposed patent. Furthermore, with respect to the process claims, it held that the claimed molar ratio of AS to AN of about 0.9 : 1 to about 1.1 : 1 was neither disclosed in nor obvious from documents D8 or D9.

V. The opponent's (appellant's) notice of appeal was received with letter dated 20 February 2007. The statement of grounds of appeal, dated 27 April 2007, was accompanied by the following new documents:

D11: Nitrogen No. 53, May/June 1968, pages 27 to 30:  
*"Ammonium Sulphate Nitrate Production- Review of commercially available processes"*

D12: GB-A-1 005 166

D13: Ullmanns Encyklopädie der technischen Chemie, 1955 Edition, vol. 6, Urban und Schwarzenberg, München - Berlin; pages 121-122

D14: The Condensed Chemical Dictionary, 7<sup>th</sup> Edition, Reinhold Publishing Corp. New York, USA, page 243

D15: WO-A-81/01 704

VI. The observations of the patentee (respondent) were received with letter dated 26 September 2007. Also submitted were:

D16: E.P. Perman and W. J. Howells: *"The Properties of Ammonium Nitrate. Part VI. The Reciprocal Salt*

*Pair Ammonium Nitrate and Potassium Sulphate*,  
J. Chem. Soc. 123 (1923), pages 2128 to 2134

D17: E. Jänecke et al.: "*Über das System  $NH_4NO_3$  -  $(NH_4)_2SO_4$  -  $H_2O$* ", Zeitschrift Anorg. Allg. Chem. 160 (1927), pages 171 to 184.

VII. Oral proceedings were held on 5 February 2010 in the absence of the appellant, who had previously informed the board that it would not attend. The respondent filed as an auxiliary request a new set of claims which differed from the set of claims as granted only in that claim 20 was deleted.

VIII. The arguments of the appellant may be summarized as follows:

The teaching of documents D3, D7, D8, D9, D11 and D12 showed that the ammonium sulfate nitrates as defined in claims 1 to 5 were inadvertently disclosed by the fertilizer industry since these compositions were merely in a state close to the thermodynamically stable state of salts resulting from melting and solidifying AN and AS at a molar ratio of around 1:1. The long known optimum mixture of 40 weight-% AN and 60 weight-% AS corresponded to a molar ratio of 0.91: 1 and was widely used in the fertilizer industry because it was below the nitrogen content limit allowable for a safe and non-explosive compound.

Even if claims 1 to 5 were found novel, they lacked inventive step having regard to D3 in combination with D8. D3 motivated a skilled person to employ the Kaltenbach process disclosed in D8 for producing

ammonium sulfate nitrate fertiliser. According to said process a molar ratio of AN/AS 1:1 was used so that the claimed product was obtained. The process claimed in claim 6 or 7 was very similar to the said Kaltenbach process of D8 which showed all the features except the ratio of AN/AS of about 1:1. However, this particular ratio of AN/AS had been widely used in the industry.

In the alternative, D3 also motivated a skilled person to modify the production method for granulated ammonium sulfate nitrate fertiliser disclosed in D12 in such a manner that fertiliser granules having the claimed compositions were obtained.

IX. The respondent rejected the appellant's arguments on novelty and inventive step.

None of D1 to D4 qualified as the closest prior art in view of the fundamental difference between the claimed composite materials and the granulated mixtures disclosed in D1 to D4.

A possible starting point for assessing inventive step were the granulated fertilizer products commercially available from BASF and Fertiberia prior to the priority date of the opposed patent. These products were granulated mixtures of AS, the 1:2 double salt and the 1:3 double salt. Each of these products contained a significantly lower amount of the 1:2 double salt than that required by the claimed invention, and a significantly higher amount of the 1:3 double salt than that required by the claimed invention.

According to the respondent, the problem to be solved



by the opposed patent consisted in providing an ammonium sulfate nitrate product having a good balance between the properties of nutrient value, safety (in terms of sensitivity to detonation) and stability (in terms of sensitivity towards moisture, resulting in caking).

Evidence that said problem was successfully solved by the composite material of claims 1 and 2 was to be found in particular in Examples 39 to 41 of the opposed patent.

The cited prior art provided no motivation that would lead the skilled person to move from a simple, granulated material towards a completely different material in the form of a composite material having the claimed proportions of AN and the 1:2 and 1:3 double salts.

There was no evidence in D8 that the Kaltenbach process resulted in a double salt. The molecular ratio of AS/AN 1:2 disclosed in D8 fell outside the ratio claimed in claim 6 of the opposed patent and would not have resulted in the 1:2 double salt.

Finally, the contents of the phase diagram according to Figure 1 of D3, on which the appellant particularly relied, was at odds with previously published data and established evidence and should therefore be treated as speculative, if not be disregarded.

X. Requests:

The appellant requested that the decision under appeal

be set aside and the patent be revoked.

The respondent requested that the appeal be dismissed or, in the alternative, that the decision under appeal be set aside and the patent be maintained on the basis of the set of claims filed during the oral proceedings as an auxiliary request.

### **Reasons for the Decision**

1. Novelty (main request)
  - 1.1 In its appeal brief, the appellant regarded each of documents D1, D2, D3, D4, D11, D12 and D13 as novelty destroying for the subject matter of claims 1 to 5, because they at least implicitly disclosed compositions falling within the definitions of said claims. However, the board does not accept the appellant's arguments, for the following reasons.
    - 1.2 Document D1 discloses the existence of 1:2 and 1:3 double salts of AS and AN and the conditions under which such double salts, together with a surplus of AS, precipitate (solidify) from a solution or melt. However, as correctly pointed out by the respondent, these salts form a mixture of crystals, not a composite material, as required by the opposed patent. The appellant maintained, however, that the claims of the opposed patent covered also simple mixtures of crystalline salts, such as described in documents D1 to D4. The appellant referred to various definitions of the term "composite" in support of its view.

- 1.2.1 Firstly, document D5 (page 377, left hand column), defined a "composite" simply as a material made up of various parts or elements, especially made of recognizable constituents.

During the appeal procedure, the appellant secondly relied on document D14, which defines a "composite" as a *"structural material composed of a mixture or combination on a macro scale of two or more components that differ in form and/or composition and that are essentially insoluble in each other"*. Examples are, inter alia, *"particulate composites, composed of particles with or without a matrix"*. D14 states that the *"behavior and properties of a composite are determined by the form and structural arrangement of the components, their composition, and any interaction between the components."*

Thirdly, the appellant furthermore referred to D12, using the term "mash" for a weakly alkaline reaction mixture of various salts such as ferric hydroxide, magnesium nitrate and ammonium sulfate before prilling (see example 1).

- 1.2.2 However, according to the definition given in Kirk-Othmer (D10), *"composites are combinations of two or more materials present as separate phases and combined to form desired structures..."*. The term "composite" as used in Kirk-Othmer assumes *"that the average dimension of the largest single homogenous geometric feature, in at least one direction, is small relative to the size of the total body in that direction; in addition, it assumes that the dimensions of the minor constituent phase are sufficiently large so that its characteristic*

*properties are substantially the same as if it were present in bulk" (D10, page 307, right hand column, first, second and third paragraphs).*

- 1.2.3 The board is of the opinion that reference D10 carries considerably more authority in the field of industrial chemistry than reference D5, so that its more precise definition of a composite material should be followed.

As regards D14, the board considers that the definition of a composite presented therein at least implicitly calls for a structural arrangement of the constituents, so that random mixtures of crystals are excluded.

In document D12 there is, in the board's view, no specific relation to a composite, so that it bears no relevance for the present case.

- 1.2.4 Furthermore, the patent itself states in paragraph [0025] that *"the composite material of the invention consists of small ammonium sulphate crystals imbedded in a matrix of other constituents. The composite material of the invention is to be distinguished from a mixture of free particles."* This definition also requires the presence of a certain structural arrangement in the form of a matrix and embedded crystals, for a material to qualify as a composite.

- 1.2.5 In the light of the foregoing, the board concludes that a mere random mixture of separate crystals fails to meet the minimum criteria for a composite according to the definitions given in the patent itself and in the relevant reference sources. For this reason alone, document D1, not disclosing such a composite, cannot

anticipate the claimed composites.

- 1.3 Document D1 also fails to disclose the proportions of the said salts, as claimed in claims 1 and 2 of the opposed patent.

Therefore, D1 does not anticipate the subject matter of claims 1 to 5.

- 1.4 Document D2 is concerned with crystallographic studies on 2:1 AS-AN and 3:1 AS-AN double salts. D2 discloses at page 77, middle column, that the 2:1 double salt was found in fertilizers in admixture with AN and AS. D2 does not disclose composites of the said salts nor their respective proportions in the fertilizers, as defined in claims 1 and 2 of the opposed patent.

- 1.5 Document D3 is a study on *"Caking and Degradation of Granular Compound Fertilizers containing Nitrates and Sulfates"*. It contains a phase diagram of the  $\text{NH}_4\text{NO}_3$  -  $(\text{NH}_4)_2\text{SO}_4$  system (Figure 1). On page 2-2 it is reported that when a mixture of two moles of  $\text{NH}_4\text{NO}_3$  and one mole of  $(\text{NH}_4)_2\text{SO}_4$  was heated to  $180^\circ\text{C}$  and cooled to room temperature, the product was a mixture of the double salt "A" =  $3\text{NH}_4\text{NO}_3 \cdot (\text{NH}_4)_2\text{SO}_4$  and  $(\text{NH}_4)_2\text{SO}_4$ , which reacted slowly at room temperature to form the double salt "B" =  $2\text{NH}_4\text{NO}_3 \cdot (\text{NH}_4)_2\text{SO}_4$ . The formation of "B" was accelerated by moisture.

- 1.5.1 The board notes that the appellant itself admits that D3 itself fails explicitly to disclose the compositions as defined in any of claims 1 to 5 of the opposed patent (grounds for appeal, page 2, first paragraph). The board cannot see an implicit disclosure, either. D3

does not, therefore, affect the novelty of claims 1 to 5.

1.5.2 The appellant asserted that D3 showed that, in practice, anyone melting AN and AS salts in a molar ratio of around 1:1 and solidifying the melt, obtained a solid phase initially containing some AS and more or less of the double salts AS-AN 1:2 ("B") and AS-AN 1:3 ("A"), depending on the process conditions, temperature and moisture. Any thus formed AS-AN 1:3 double salt then decomposed to form the thermodynamically stable compound AS-AN 1:2, a transition which was, according to D3, accelerated by moisture. Thus, any fertilizer made from mixtures of AN and AS in a molar ratio of about 1:1 would sooner or later upon storage "inadvertently" reach compositions falling within the definitions of claims 1 to 5 of the opposed patent. The appellant asserted that the industry had for many years manufactured such fertilizers by granulating or prilling melts of about 40 weight-% AN and 60 weight-% AS (corresponding to a molar ratio AN:AS of 1:0.9).

1.5.3 However, even if one accepted that fertilizers made from melts consisting of about 40 weight-% AN and 60 weight-% AS (corresponding to a molar ratio AN:AS of 1:0.9) were usual in the art, it does not follow from D3 that such fertilizers, upon storage, decomposed to a mixture of about 14 wt.% to about 35 wt.% ammonium sulfate ( $(\text{NH}_4)_2\text{SO}_4$ ), about 60 wt% to about 85 wt.%  $(\text{NH}_4)_2\text{SO}_4 \cdot 2 \cdot (\text{NH}_4\text{NO}_3)$  (double salt "B") and up to about 5 wt.% in combined total  $(\text{NH}_4)_2\text{SO}_4 \cdot 3(\text{NH}_4\text{NO}_3)$  double salt ("A") and ammonium nitrate ( $\text{NH}_4\text{NO}_3$ ).

Moreover, such a hypothetical mixture of "A", "B", AS

and AN would not, or not necessarily, form a composite, as required by claims 1 or 2, but a mixture of separate crystals.

- 1.6 Document D11 discloses *inter alia* the so-called Fisons process for making ammonium sulfate nitrate fertilizers. This process is characterized by the addition of considerable quantities of ammonium bisulfate (ABS) to the AN/AS mixture, leading to a melting point of as low as 80 °C, but normally between 90 and 120°C. The molar ratio of AN to ABS is between 1:1 and 2.2:1. In the process, (all of) the ABS is converted (ammoniated) to AS. The appellant asserted that a product as claimed in the opposed patent was formed, in view of the process temperature falling in a range where, according to the phase diagram of Figure 1 of D3, the stable phases of "B" plus AS exist.

Furthermore, the appellant cited D12, example 4, as showing a prilled ammonium sulfate fertilizer made from prilling a melt at 180°C of AN:AS at a ratio of 1.06:1. According to the appellant's arguments, said product "*obtained a similar composition as defined in claims 1 to 5*", thereby depriving said claims of novelty.

D13 was cited by the appellant to show that it was known in 1955 to make compositions with a total nitrogen content of 26% starting from a reactant mixture of about 40 weight-% AN and 60 weight-% AS. The appellant argues that said starting mixture resulted in a fertilizer product consisting of a crystal mixture of the AS/AN 1:2 double salt and some excess AS.

However, even if one accepts to the benefit of the

appellant that D11, D12 and D13 disclosed, at least implicitly, crystal mixtures of the AS/AN 1:2 double salt plus some excess AS, these documents do not disclose the respective amounts of these constituents, as claimed in the opposed patent. Furthermore, said crystal mixtures are distinguished from the composite material as claimed, as explained above.

For these reasons, D11 to D14 do not anticipate the claimed subject matter, either.

- 1.7 In the opposition proceedings, the opponent (appellant) cited document D4 as novelty destroying for the subject matter of claims 1 to 4. The opposition division rejected the opponent's assertions in the opposed decision and no further arguments were brought forward during the appeal procedure in this respect. Therefore, the board need not go into further detail, save the remark that the board is unable to detect all the claims' features in D4.
- 1.8 The appellant did not bring forward any novelty arguments against process claims 6 to 19 or against the independent product-by-process claim 20; the opposition division accepted these claims as being novel having regard to the prior art of D1 to D9. The board is satisfied that the subject matter of these claims is not anticipated by any of documents D1 to D15.
- 1.9 The board concludes that claims 1 to 20 in accordance with the main request meet the novelty requirement of Article 54(1), (2) EPC.



2. Inventive step

2.1 According to paragraph [0002], the opposed patent relates to ammonium sulfate nitrate composites useful as fertilizers having desirable levels of nitrate ions, superior stability against detonation, higher density, greater resistance to moisture, and a method for their manufacture.

2.2 In its appeal brief, the appellant started from document D3 as the closest prior art. The appellant argued lack of inventive step of claims 1 to 5 having regard to D3 in combination with D8, D11 or D12.

The board notes that D3 is concerned with caking behaviour of granular compound fertilizers containing nitrates and sulfates and addresses neither detonation safety nor moisture resistance or density.

To the benefit of the appellant the board takes document D3 as the starting point for assessing inventive step.

2.3 The technical problem underlying the opposed patent in the light of D3 may be defined as providing an ammonium sulfate nitrate product having a good balance between the properties of nutrient value, safety (in terms of sensitivity to detonation) and stability (in terms of sensitivity towards moisture, resulting in caking) (see also paragraph [0017] of the opposed patent). This definition corresponds to the respondent's formulation of the technical problem.

2.4 As a solution to this technical problem the patent in suit proposes an ammonium sulfate nitrate according to claim 1 or claim 2 characterized in that the ammonium sulfate ( $(\text{NH}_4)_2\text{SO}_4$ ) content is about 14 wt.% to about 35 wt.%, the content of the double salt  $(\text{NH}_4)_2\text{SO}_4 \cdot 2(\text{NH}_4\text{NO}_3)$  is about 60 wt.% to about 85 wt.% double salt and the combined total content of  $(\text{NH}_4)_2\text{SO}_4 \cdot 3(\text{NH}_4\text{NO}_3)$  double salt and ammonium nitrate ( $\text{NH}_4\text{NO}_3$ ) is from 0 to about 5 wt.%.

2.5 In fact, the opposed patent (paragraphs [0015] and [0025]) contains undisputed evidence that double salts are safer against explosion, as they provide intimate dilution of the ammonium nitrate at a molecular level. Experimental data had been obtained (although not reported in the patent) showing that the 1:3 double salt released more energy on decomposition than the 1:2 salt. Example 39 (Table VIII) of the patent revealed that the ammonium sulfate nitrate of the invention exhibited a higher onset temperature (220 to 236°C) for spontaneous and energetic decomposition than both the 1:3 double salt (210°C) and AN (205 to 210°C). The appellant had not denied that the parts of the problem relating to nutrient value and stability (in terms of sensitivity towards moisture) had been solved by the claimed composites.

As to the nutrient value, it was known according to the respondent that the nourishing property was due to the nitrate content of the composite material.

As regards sensitivity to moisture, the respondent plausibly argued that caking problems could be avoided by adjustment of the water content. It is also shown in example 41 that the critical humidity level (*i.e.* the

level at which the material begins to absorb moisture from the atmosphere) for the ammonium sulfate nitrate of the invention at 30°C was 75% R.H. The claimed composite material is thus less sensitive to moisture than ammonium nitrate (60% R.H.), and similar to ammonium sulfate (80% R.H.) in its moisture sensitivity. The balance between these properties (nutrient value, sensitivity to detonation and sensitivity to moisture) was struck where safety was assured (*cf.* patent in suit, page 3, lines 55 to 57).

The board is therefore satisfied that the technical problem as defined above was successfully solved.

- 2.6 Finally, it remains to be decided whether or not the proposed solution to the technical problem, namely the ammonium sulfate nitrate composite material according to claim 1 or claim 2, is obvious in view of the cited prior art.
- 2.7 The appellant argued that it was known from D8, page 34, left hand column, that the formation of prills or granules minimized the problems of caking and insufficient hardness. D3 taught that solidification of a melt of molar ratio of about 1:1 of AN and AS yielded a composite phase of mostly AS/AN 1:2 double salt, excess AS and minor amounts of AN and AS/AN 1:3 double salt. The latter would be gradually transformed to the 1:2 double salt under the influence of moisture. Thus the skilled person found motivation in D3 for using a process route such as the so-called Kaltenbach process of D8 using a molar ratio of AN/AS of about 1:1, and prilling the product, in order to solve the problem posed.

2.8 The board observes that the Kaltenbach process, described on page 35 of document D8, was designed for the manufacture of the double salt ammonium sulfate/ ammonium nitrate in a molar ratio of 2:1, starting from an aqueous slurry of 95% AN and 5% water and ground ammonium sulfate. The use of additives to lower the melt temperature prior to prilling is usually avoided. A prilled product is obtained having a moisture content of between 0.3 and 0.4%.

Ammonium sulfate nitrate is generally described in D8 (page 34, left hand column, middle paragraph) as having a smaller explosion risk than ammonium nitrate and a higher nitrogen content than ammonium sulfate. It was also less hygroscopic than ammonium nitrate, although caking of the pure product may still occur. A material with an anti-caking nature and sufficient hardness was usually obtained through the additives and the formation of granules or prills.

In the board's view the appellant's arguments are flawed in that, according to D8, the Kaltenbach process starts from a molar ratio of AN/AS of 2:1, rather than from the significantly different ratio of AN/AS of 1:1. The molar ratio of AN/AS of 2:1 is maintained throughout the process *"in order to enhance the prilling operation and the storage characteristics of the finished product"* (page 35, left hand column, penultimate paragraph). Therefore, it is not seen how this different material could yield a composite phase of mostly AS/AN 1:2 double salt, excess AS and minor amounts of AN and AS/AN 1:3 double salt, according to Figure 1 of D3, which forms from melts having a 1:1

molar AS/AN ratio (see point 1.5.2 above).

Moreover, there is no teaching in either D3 or D8 that a composite phase consisting predominantly of the 1:1 double salt was less sensitive against detonation, and hence there was no incentive to examine such phases in view of the problem posed.

The board concludes that it would not have been obvious in the light of D3 and D8 to provide a non-explosive ammonium sulfate nitrate composite material comprising a major proportion of about 60 wt% to about 85 wt.%  $(\text{NH}_4)_2\text{SO}_4 \cdot 2(\text{NH}_4\text{NO}_3)$  double salt and to limit to about 5 wt.% the combined total of  $(\text{NH}_4)_2\text{SO}_4 \cdot 3(\text{NH}_4\text{NO}_3)$  double salt and ammonium nitrate  $(\text{NH}_4\text{NO}_3)$ , as indicated in the claims, in order to solve the part of the problem relating to detonation safety. It is also not obvious in the light of the cited prior art that a composite material might be safer than a crystal mixture.

2.9 The appellant also asserted that D3 motivated a skilled person to modify the production method for granulated ammonium sulfate nitrate fertiliser disclosed in document D12 in such a manner as to obtain fertiliser granules with the claimed compositions.

However, the appellant's assertion is not supported by reasons as to why the skilled person should combine D3, and in particular a specific composition shown in a phase diagram in D3, with process features known from D12. The teaching of D3 (see conclusion on page 2-5, last paragraph) is to decrease the amount of the 1:2 double salt ("B") to prevent degradation and caking. Applying this teaching to D12 would not lead to the

- claimed products, which contain, in the form of a composite material, a major proportion of the 1:2 double salt ("B").
- 2.10 For these reasons, the subject matter according to claims 1 and 2 involves an inventive step.
- 2.11 Claim 6 is directed to a method for the production of an ammonium sulfate nitrate composite material.
- 2.12 It appears from the reasoning of the appellant that D8 is considered to represent the closest state of the art. The board also takes D8 as the starting point for assessing inventive step.
- 2.13 The problem underlying the patent in suit in the light of D8 in so far as claim 6 is concerned is the provision of a further method for producing an ammonium sulfate nitrate composite material.
- 2.14 As a solution to this problem the patent in suit proposes a method according to claim 6 characterized in that
- (a) materials comprising ammonium sulfate particles, ammonium nitrate and water are charged to a melting device, wherein the molar ratio of ammonium sulfate to ammonium nitrate is about 0.9:1 to about 1.1:1 and the water is more than 2 wt.% to about 10 wt.% of the charged materials;
  - (b) the ammonium nitrate is melted and at least a portion of the ammonium sulfate particles is dissolved at a temperature of about 180°C to about 210°C;
  - (c) the charged materials are reacted at a temperature of about 180°C to about 210°C; and

(d) the product is solidified at a cooling rate of at least about 100°C/min."

- 2.15 The 36 examples (of which 14 comparative examples) displayed in Tables IV and V of the opposed patent show that the problem defined under point 2.13 above has been plausibly solved.
- 2.16 It remains to be decided whether or not the proposed solution to the technical problem, namely the method for the production of an ammonium sulfate nitrate composite material according to claim 6, is obvious in view of the cited prior art.
- 2.17 The appellant's arguments concerning lack of inventive step of process claim 6 suffer from the same inconsistency as above, in that the Kaltenbach process applies a molecular ratio of AN/AS 2:1 (see page 35, left hand column), whereas the appellant's arguments concerning D3 assume a ratio of AN/AS of 1:1. Although a prilling temperature of 160°C to 182°C may be conventional for ammonium sulfate nitrate (D12, column 2, lines 43, 44), D8 itself does not disclose a reaction temperature of the charged materials of about 180°C to 210°C, as required by claim 6.
- 2.18 Finally, the combination of document D3 with document D12 cannot suggest the claimed process, either. D12 discloses a process for making prilled ammonium sulfate nitrate having improved storage stability by adding to the melt ("mash") an amphoteric oxidic compound (ferric oxide) and a weakly alkaline compound. In example 4 thereof, a starting ratio of AN/AS of 1.06:1 and a prilling temperature of 180°C are employed. The

appellant argued that it would have been obvious to add a small amount of water to that charge in order to accelerate the transformation of the 1:3 double salt to the 1:2 double salt, as taught in D3. However, to do so would be contrary to the express teaching of D3 which suggests, as noted above, decreasing the amount of this 1:2 double salt ("B") to prevent degradation and caking. Therefore, the board does not accept the appellant's arguments.

It follows that the process of claim 6 involves an inventive step.

- 2.19 The appellant did not bring forward objections or arguments concerning the subject matter of product-by-process claim 20. In view of this, the board limits itself to remarking that the product resulting from the process conditions recited in claim 6 fulfils the requirements of claim 1. This view is supported by Tables IV, V and VI of the patent in suit displaying 36 examples, of which 14 comparative examples, which demonstrate the influence of the critical molar ratios and of the water content on the final composite ammonium sulfate nitrate.

The reasoning regarding the non-obviousness of the subject matter according to claim 20 is the same as that outlined under points 2.7 to 2.9. Hence the subject matter of claim 20 also involves an inventive step.

- 2.20 Claims 3 to 5 and 7 to 19 derive their patentability from independent claims 1, 2 and 6, respectively, from which they depend.



2.21 In conclusion, the claims of the main request meet the requirement of Article 56 EPC.

2.22 As the main request is allowable, there is no need to consider the auxiliary request.

## **Order**

**For these reasons it is decided that:**

The appeal is dismissed.

The Registrar

The Chairman

C. Eickhoff

G. Raths